

Exhibit 14.12

United States' Motion to Enter Consent Decree,
United States v. Alden Leeds, Inc. et al., Civil Action No. 22-7326 (D.N.J.)

EXHIBIT A-41

Appendix A to OxyChem's Comments in Opposition to Proposed Consent Decree,
United States v. Alden Leeds, Inc., et al., Civil Action No. 2:22-cv-07326 (D.N.J.)

Process for 2,4,5 Trichlorophenol

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MICROFILMED

MAR 30 1951

2,4,5 TRICHLORPHENOL

Krebs
8-24-48

Manufactured at
GIVAUDAN-DELAWANNA INC.
Delawanna, N. J.

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Process for 2,4,5 Trichlorophenol

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Date 8-24-48 Original Process H. G. Krebs

A handwritten signature in cursive script, appearing to read "H. G. Krebs", written in dark ink.

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CURRENT PROCESS

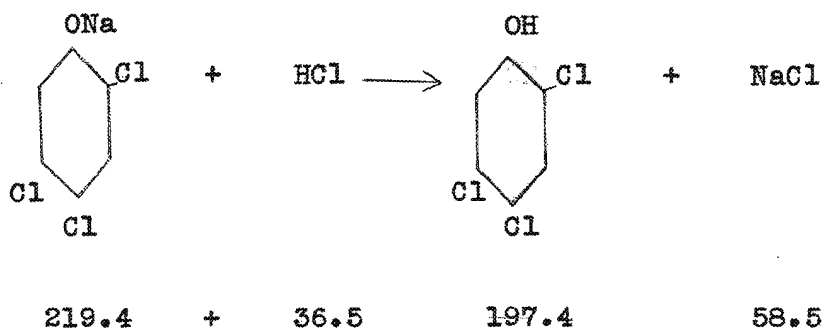
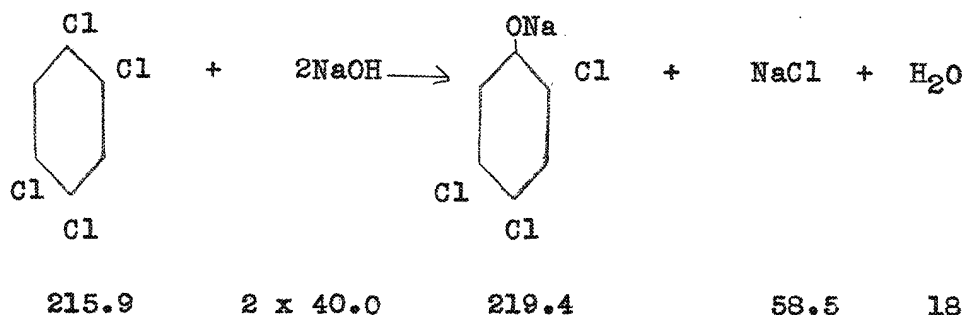
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OBSOLETE SECTIONS

SECTION I

CHEMISTRY OF PROCESS

2,4,5 Trichlorophenol is made by hydrolyzing 1,2,4,5 tetrachlorobenzene with an excess of caustic soda dissolved in ethylene glycol at a temperature of 170 to 175°C. After the reaction the batch is neutralized with muriatic acid and the sodium chloride which is precipitated is removed by filtration. The filtrate is diluted with water and the trichlorophenol is extracted with benzol. The benzol extract is washed with water and the benzol is removed by distillation. The crude trichlorophenol is vacuum distilled, and a product with a congealing point of 63°C is obtained. The ethylene glycol which is recovered by fractionation is reused in the process.



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The theoretical weight yield based on tetrachlorbenzene is $91\frac{1}{2}\%$. With the fair grade of tetrachlorbenzene employed an actual weight yield of 70 to 76% has been obtained in the plant.

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SECTION II

RAW MATERIALS

1,2,4,5 Tetrachlorbenzene:

Melting Point: 136°C minimum

Purchased from Hooker Electrochemical Co. Received in M3208 leverpak containers holding 200 pounds.

Caustic Soda Flakes:

Purity: Minimum 96% as NaOH

Purchased from Sergeant Pulp & Chemical Co., Inc. Received in department in steel drums containing 400 pounds of flakes.

Ethylene Glycol:

Specific Gravity 25/25°C: 1.1115 to 1.1130

Boiling Range: 195 to 205°C

Purchased from Dow Chemical Co. and Carbide & Carbon Chemicals Corp. Received in department in steel drums containing 500 pounds.

Recovered Ethylene Glycol:

As recovered in this process.

Specific Gravity 25/25°C: 1.110 minimum

Muriatic Acid 20° Baumé:

Purity: 31.5 to 35.2% as HCl

Specific Gravity 25/25°C: 1.1528 to 1.1709

Purchased from A. H. Mathieu & Co. and Riches Nelson Co. Received in department in carboys containing 115, 118 or 131 pounds of acid.

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Isopropyl Alcohol 99%:

Specific Gravity 25/25°C: 0.783 to 0.787

Distilling Range: Total range not more than 1.5°C including the temperature 82.5°C.

Non-Volatile Matter: Maximum 5 mg. per 100 ml.

Purchased from Standard Alcohol Co., Standard Oil Co., Shell Chemical Co. and Enjay Co., Received in tank cars. Delivered to department in 55 gallon drums containing 300 pounds.

Recovered Isopropyl Alcohol:

As obtained by fractionating weak isopropyl alcohol recovered in this process.

Purity: 85% by weight minimum

Specific Gravity 25/25°C: 0.820 maximum

Benzol:

Specific Gravity 25/25°C: 0.875 to 0.877

Congeaing Point: 5°C minimum

Distilling Range: Not more than 1°C including the temperature 80.1°C

Purchased from Stoney Mueller Inc., Calco Chemical Div., and Jones & Laughlin Steel Co. Received in tank cars. Delivered to department in 55 gallon drums.

Benzol Recovered:

As recovered in this process.

Specific Gravity 25/25°C: 0.870 to 0.877

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Soda Ash:

Purity: Minimum 98% as Na_2CO_3

Purchased from A. H. Mathieu, Solvay Sales and Solvay
Process. Received in department in paper bags contain-
ing 100 pounds.

#4619 150 gal. half jacketed steel tank with bolted cover, equipped with thermometer well and jacketed vent pipe leading through the roof. This tank is used to vent residual 20 lb. pressure from hydrolysis kettle at the end of the reaction.

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SECTION III

EQUIPMENT

Building 54

#8454 Stainless Steel Reaction Kettle; capacity 250 gallons; gas fired, anchor type agitator driven at 52 RPM through #8412 Nettco speed reducer by 2 horsepower motor #8673; stainless steel cooling coil with 14 square feet of cooling surface; top of kettle equipped with 6" charging hole, 1 $\frac{1}{4}$ " drum charging leg with flexible hose, thermometer well for 0 to 300°C Motoco thermometer and Gotham recorder, steam jacketed vent line, combination vacuum, vent and air lines, cooling coil inlet and outlet; 2" relief valve set at 25 pounds, 0 to 60 pound pressure gage; 2" outlet on bottom of kettle closed by 2" nickel plug cock connected to 1 $\frac{1}{2}$ " stainless steel blow line to building 60 and to drain cock. Used for hydrolysis reaction.

#4619 —

#8543 Gotham single pen recorder 0 to 300°C. Used to record temperature in hydrolysis kettle #8454.

#8484 Air Hoist; Ingersoll-Rand; capacity 500 pounds. Used to charge hydrolysis kettle #8454.

#8868⁷ Monel High Vacuum Still; capacity 186 gallons, gas fired, oil jacket, equipped with 1 $\frac{1}{4}$ " drum charging leg with flexible metallic hose; thermometer well in oil jacket for 0 to 300°C Motoco thermometer; thermometer well in kettle for bulb of Gotham recorder; 15 pound steam traced relief valve and vent, 18" diameter packed monel column filled to a height

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of 10 feet with 1" porcelain Raschig rings; monel coil dēphlegmator with 5.5 sq. ft. of cooling surface at top of column. Used to distill the crude trichlorophenol.

#8824 Monel Coil Condenser - 36 square feet of cooling surface; cooled by recirculated warm water at 70°C; inlet connected by gooseneck to top of 18" diameter packed column, outlet to either monel receiver #8884 or #8885. Used to condense trichlorophenol vapors from monel still #8868.

#8884 & #8885 Monel Water Jacketed Receivers; capacity 25 gallons each; jackets supplied with water at 70°C from water recirculation system; steam heated level gage glass on side; top connections to condenser #8824, to 0 to 30" vacuum dial type gage, to Zimmerli gage, to ^{jacketed} vacuum trap which in turn is connected to coarse, medium and high vacuum supply; $\frac{3}{4}$ " drain cock on bottom. Used to receive trichlorophenol condensate.

#8691 Worthington Iron Centrifugal Pump; capacity 25 GPM; $1\frac{1}{2}$ " inlet connected to hot water tank automatically controlled at 70°C by TR-21 Sarco regulator; outlet connected to jackets of monel receivers, monel condenser and to monel dēphlegmator coil through 0 to 5 GPM rotameter. 0 - 100°C Motoco thermometers at inlet and outlet of dēphlegmator coil, Used to circulate warm water through monel condenser #8824, dēphlegmator coil, and jackets of monel receivers #8884 & #8885.

#8772 Gotham Recorder - three pen 30 to 240°C. Used to record still, vapor and distillate temperatures of monel still.

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#8728 Glycol Recovery Still; steel; capacity 600 gallons; 100 pound code jacket connected to steam supply and cooling water; top connected to #8787 30" diameter packed column, to drum charging line, to 275 gallon storage tanks #5640, 5641 5642 & 9029; thermometer well for one bulb of three pen Gotham recorder and o to 200°C Motoco thermometer; drain on bottom to sewer and to residue drum. Used to distill the weak ethylene glycol.

#8787 Packed Column; steel; diameter 30 inches; height 11 feet; filled with 1" porcelain Raschig rings to a height of 9½ feet; reflux distributor at top; two wall collectors and distributors; bottom connected to top of still #8728; top connected by gooseneck to condenser #8767; side connection at top for liquid reflux connected to reflux proportioner. Used to fractionate the ethylene glycol.

#8767 Steel Water Tube Condenser; 15" diameter x 56" length; 60.6 sq. ft. cooling surface; 1½" water inlet and outlet and condensate connections; vapor inlet connected to top of column #8787; distillate outlet connected ^{to} reflux proportioner to liquid cooler. Used to condense vapors from column #8787.

#8670 & 8671 Steel Vacuum Receivers; capacity each 150 gallons; 31½" inside diameter; distillate inlet at top from liquid cooler; top connected to coarse, medium and fine vacuum supply; level gage glass on side; bottom outlet to drum line running to outside of building, to drum line inside of building, and to 750 gallon steel tank. Used to receive benzol, isopropyl,

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alcohol, water and ethylene glycol fractions from liquid cooler.

Panel Board for glycol still; equipped with four 0 to 150°C, Motoco thermometers for indicating temperature of reflux flowing to column, of condensate from tubular condenser #8767, of water to and from tubular condenser; also Zimmerli gage for indicating absolute pressure at top of column; mercury U tube gage for indicating pressure drop in column; Gotham temperature recorder #8636 mounted in center of board.

#8599 Horizontal Steel Tank; 750 gallon capacity; inlet at top from receivers #8670 and 8671; level gage glass on side; bottom connected to Westco pump #8561. Used to receive water fraction from glycol recovery still. 3'11½" I.D.

#8561 Westco Turbine Pump; all bronze; capacity 25 GPM. Used to pump recovered water from 750 gallon storage tank to building 60.

#8636 Gotham Temperature Recorder; three pen; range 0 to 200°C. Used to record temperature of still, vapor and distillate of ethylene glycol recovery still #8728.

#8656 Nash Hytor Vacuum Pump; Size A1-673; ½" water inlet; 1½" vacuum inlet; 1½" discharge; driven at 1750 RPM by 3 horsepower motor #8891. Used to supply coarse vacuum for glycol recovery still #8728 for charging and distilling light fractions.

* This kettle has an 8" galvanized vent pipe leading outside the building to remove fumes while the hot batch is being blown over.

Building 60

#6220 Kettle; stainless steel; rated capacity 250 gallons; jacket connected to steam and cooling water; turbine type agitator driven at 190 RPM through #8412 speed reducer by 2 horsepower motor #8673; top of kettle fitted with 6" hand hole, thermometer well for 0 to 200°C Motoco thermometer, vent and vacuum connection, 1½" inlet from hydrolysis kettle #8454 in building 54, ½" Saran muriatic acid connection; bottom connection to drain cock and to Westco pump #8449. Used to cool and neutralize glycol slurry from hydrolysis kettle. This kettle is actually too small for this process. *

#8858 Sigma Pump, Model T-6, pulsating rubber tube type pump; capacity ½ gallon per minute; driven at 280 RPM through pulleys by ¼ horsepower motor #8254; inlet from acid carboys outside of building; outlet connected to Saran tubing to neutralizer #6220. Bypass on pump so that acid can be fed to neutralizer by vacuum without the use of pump. Used to feed last portion of muriatic acid at a slow rate to neutralizer #6220.

#8449 Westco Turbine Pump; bronze; capacity 25 gallons per minute driven by 1½ horsepower motor #8250. Used to pump slurry from neutralizer #6220 to filter press #4653.

#4653 Shriver Filter Press - 24"; cast iron head; 17 - 1" thick bronze frames; bronze plates; through washing; feed inlet connected to Westco pump #8449, wash port connected to isopropyl alcohol tank #8565; filtrate ports connected to 1¾" pipe line to extraction tank #6763 and to return line to

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neutralizer #6220; air connection for blowing press. Number 8 Jabeeco cotton duck filter cloth is used as the filter medium. This press will be replaced with an all bronze press. Used to remove sodium chloride from neutralizer slurry.

#8565 Steel Tank; capacity 75 gallons, $23\frac{1}{2}$ " inside diameter x 38" straight height; top provided with $1\frac{1}{4}$ " charging line, 1" coarse vacuum and vent connection; level gage on side equipped with spring closing valve, 1" bottom outlet connected to wash port of filter press #4653 and also to suction line of filter press #4653. Used as a storage tank for isopropyl alcohol.

#6763 Lead Lined Extraction Tank; capacity 1130 gallons; 36" diameter 10 bladed hard lead turbine type agitator driven at 71 RPM through Nettco speed reducer #6770 by $7\frac{1}{2}$ HP motor #8538; thermometer well for 0 to 150°C Motoco thermometer; top provided with $1\frac{1}{4}$ " inlet from filter press, 1" water line connected to water meter for either fresh or recovered water, $1\frac{1}{2}$ " vent, manhole cover with 6" charging hole, $1\frac{1}{4}$ " feed connection from steel tank #6235; monel decanter arm mounted in side of tank, 2" bottom outlet connected to $1\frac{1}{4}$ " drain cock, $1\frac{1}{4}$ " drum line and $1\frac{1}{4}$ " brass line to steel neutralizer #6219. Used to dilute glycol filtrate from press #4653 and to extract the trichlorophenol from the dilute glycol by means of benzol.

#6235 Steel Tank; capacity 250 gallons, $35\frac{1}{2}$ " inside diameter x 56" straight side; $1\frac{1}{4}$ " top connection from Westco pump #8448, $1\frac{1}{2}$ " overflow line to underground tank #8600, level

In place of #8528

#9176 500 gal. Monel metal Still; turbine agitator,
Monel heating and cooling coil, the latter equipped with
safety valve set at 50 lb. pressure.

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gage glass on side with spring closing valve, $1\frac{1}{2}$ " outlet on bottom connected to extraction tank #6763. Used to measure benzol which is added to extraction tank #6763.

#8528 Copper Agitated Still; capacity 350 gallons, copper heating and cooling coil with 13.4 sq. ft. of surface; 20" diameter 8 bladed bronze Nettco turbine agitator driven at 142 RPM through Nettco Speed reducer #5205 by 3 horsepower motor #8518; top equipped with gooseneck connection to condenser #5713, safety valve set for 10 pounds per sq. inch, 6" hand hole opening, thermometer well for 0 to 200°C Motoco temperature indicator, drum charging line, vacuum connection, suction line to decanter of lead lined extraction tank #6763; bottom drain to drum or sewer. Used to receive extracts from extractor #6763, to wash benzol extract and to remove benzol from the crude trichlorophenol.

#5713 Copper Coil Condenser; 38 sq. ft. of cooling surface; inlet from gooseneck of copper still #8528; discharge to vacuum receiver #8526 and to underground tank #8600. Used to condense vapors from copper still #8528.

#8526 Steel Receiver; capacity 25 gallons; $17\frac{1}{2}$ " inside diameter by 24" straight side; connections on top to condenser #5713 and to coarse vacuum and vent line; level gage glass on side; bottom connection to drain valve and to underground tank #8600. Used to receive condensate from condenser #5713.

#8600 Underground Tank; capacity 750 gallons; 48" inside diameter by 96" straight side; in horizontal position; top connections for vent, fill line to surface, fill line from

condenser #5713, overflow line from tank #6235, suction line to Westco pump #8448, gage connection to remote level indicator #8647; 18" manhole; located on south side of building 60. Used to receive dry benzol condensate from condenser #5713.

#8647 Uehling Tank-O-Meter; scales 2 to 48" and 10 to 750 gallons; equipped with hand air pump. Used to measure amount of benzol in underground tank #8600.

#8448 Westco Turbine Pump; all bronze; capacity 25 gallons per minute; driven at 1750 RPM by one horsepower motor #8540. Used to pump benzol from underground tank #8600 to measuring tank #6235.

#6219 Steel Neutralizer; capacity 500 gallons; 53-3/8" inside diameter, straight length - 48"; 18" diameter 6 bladed cast iron Nettco turbine agitator driven at 90 RPM through speed reducer #8883 by 2 horsepower motor #8981; top connections for fill line from bottom of extractor #6763, drum charging line, vacuum and vent lines; bottom drain to drum, sewer and outside storage tanks #5640, 5641, 5642 and 9029. Used to neutralize dilute ethylene glycol from extraction tank #6763.

#5640, 5641, 5642 and 9029 Storage Tanks; steel; fuel oil type; each 275 gallons; 60" length by 44" height by 27" width; located outside of building 60 on North side; top of all tanks connected to common header which in turn is connected to vent; bottom of each tank connected to fill line from neutralizer #6219 and to suction line of Westco transfer

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pump #8500. Used to store dilute ethylene glycol from steel neutralizer #6219.

#8500 Westco Turbine Pump; all bronze; capacity 28 GPM; driven at 1750 RPM by one horsepower motor #8980; suction connected to bottom of storage tanks #5640, 5641, 5642 and 9029, and to bottom of neutralizer #6219; discharge connected to overhead pipe line to glycol recovery still #8728 in building 54. Used to pump weak ethylene glycol from storage tanks to ethylene glycol recovery still #8728.

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SECTION IV

MATERIAL AND LABOR REQUIREMENTS

Raw Materials

Hydrolysis

Ethylene Glycol - fresh or recovered	1,380 pounds
Tetrachlorobenzene	600 "
Caustic Soda Flakes	312 "

Neutralization

31% Muriatic Acid	approx.	575	"
99% Isopropyl Alcohol (or recovered)	"	170	"

Extraction and Washing

Benzol - fresh or recovered	1,500 pounds
Muriatic Acid	approx. 25 "

Neutralization of Extracted Dilute Ethylene Glycol

Soda Ash	approx. 30 pounds
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Labor Requirements

The department has been operated with one first class operator, two second class operators and ³four chemical laborers for the three shifts. It is recommended that all the operators be at least second class operators.

On the day shift, there are ²three operators. One in building 54, another in building 60 and the third who is the first class operator works in both buildings. During the second and third shifts there is an operator in each building. Thus a total of ⁶seven operators are required for twenty four hour

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operation.

One hydrolysis batch is made on each shift so that 3 batches are made every day.

The recovered isopropyl alcohol is rectified in another department.

SECTION V

OPERATING PROCEDURE

Hydrolysis:

Hydrolysis kettle #8454 is charged with 1,380 lbs. of either fresh or recovered ethylene glycol with vacuum. The cock on the steam heated vent line is opened and the steam to the vent line and copper tubing around the pipe connections on top of the kettle is turned on. The agitator is started and 312 lbs. of caustic soda flakes which have been previously weighed into an old tetrachlorobenzene fiber drum are charged by means of the elevator and air hoist. Then 600 lbs. of tetrachlorobenzene, which has been loosened by beating the fiber containers with a short section of $1\frac{1}{2}$ " lead lined pipe, are charged. The hand hole cover is bolted down and the gas flame is turned on full. When the batch temperature reaches 140°C , the cock in the steam heated vent line is closed. At 165°C , the gas flame is turned off completely, and at 172°C the cooling water to the cooling coil is turned on full. As soon as the temperature levels off at 175 to 176°C , the cooling water is turned off. The temperature is maintained for four hours at 175°C . A quarter inch flame on all the burners is sufficient to maintain 175°C . A pressure of approximately 20 pounds per sq. inch is obtained at 175°C and in view of the fact that the safety valve on the kettle is set for 25 pounds, this temperature should not be allowed to rise more than a few degrees above 175°C at any time. At the end of the four hour period and with the gas still on, the pressure

within the hydrolyzer is slowly vented into condensate tank #4619. The latter is cooled by circulating water in the jacket during this operation. Venting must not be done too fast since some hot vapors may go past the cooling surface and be lost from the condensate tank through the roof vent. When the pressure within the hydrolyzer has dropped to 0, the temperature of the batch is quickly brought to 185°C and kept here ten minutes, then the batch is ready to be cooled to 105°C. If the safety valve does open at any time, the vent line from the safety valve should be inspected before the next batch is started in order to insure that no blockage has occurred. In addition, this vent line should be inspected at least once a month as there is always the possibility that the safety valve has leaked slightly and has clogged the vent line. After the cock in the vent line is closed, the temperature must be watched very closely as a heat of reaction develops in the vicinity of 150 to 160°C. At the end of the heating period the batch is cooled to approximately 105°C (not below 95°C as the batch thickens if the temperature is too low) by means of cooling water to the coil. About 10 minutes before the batch is to be blown to Building 60, the plug is removed from the cross on the bottom discharge connection of the hydrolysis kettle, and the nickel cock and pipe connections at the bottom are heated with the gas torch for about 10 minutes. The plug is replaced and the batch is blown to Building 60 by means of air.

The contents of the vent tank #4619 are removed when

the batch has been blown out of the hydrolyzer. This condensate is in two layers, a lower layer containing a small amount of glycol and some dissolved halogenated benzenes and an upper oily layer consisting of liquid by-products. These are drummed up for disposal.

Neutralization:

Before the batch is blown over from building 54, the three bottom valves on neutralizer #6220 are closed and the agitator and cooling water to the jacket are turned on. After the batch has been blown, the level in the neutralizer is measured and recorded. The distance between the bottom of manhole and the top surface of the batch should be 13 inches. When the temperature of the batch falls to 75°C, neutralization of the batch is started by adding approximately 550 pounds of muriatic acid from carboys by means of vacuum. The cooling water to the jacket is used during this neutralization. If the temperature of the batch is allowed to fall to about 55°C before the acid addition is started, caking will occur around the sides and the batch will thicken so that the acid will not mix and there will be danger of the free acid corroding the top of the stainless steel neutralizer. The hand hole cover is removed and the batch is checked with congo red paper. The batch should still be alkaline. Additional muriatic acid is added at a slow rate by means of the Sigma pump #8858 until the batch is acid to blue litmus paper but not to congo red paper. A total of approximately 575 pounds of acid is required. The cooling water is turned off when the batch has cooled to 25°C.

Filtration:

While the batch is being cooled in the neutralizer, the lead lined extraction tank #6763 is charged with 50 gallons of recovered water from building 54.

As soon as the temperature of the slurry in the neutralizer has fallen to 25°C, filtration is started. During the operation of the filter press, a plastic cover is placed over the press in order to guard against any spurts of liquor from the press. In addition, the operator must wear a face shield when working around the press. The slurry is filtered in filter press #4653 set up with 17 frames. The filtrate is returned to the neutralizer until it runs clear and then it is switched over to the lead lined extraction tank #6763 after the 36" diameter agitator has been started. Filtration of the batch requires approximately 30 minutes. When the neutralizer is empty, the pipe lines and pump are flushed with two pails of drip pan isopropyl alcohol drainings obtained from the previous filtration. (If none is available, a pail of isopropyl alcohol is used.)

The filter press is then blown with air for 2 minutes into the lead lined extractor. The press is washed by gravity with either fresh or recovered isopropyl alcohol from wall tank #8565. When the alcohol appears in the sight glass in the filtrate line, the wash valve is closed and the press is allowed to soak for 5 minutes. The press is then blown for 5 minutes into the extractor. Approximately 6 inches of isopropyl alcohol (75 pounds) measured in steel tank #8565 are required for the wash.

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The damp salt cake which weighs approximately 300 pounds is removed from the press and is discarded. (The press is dumped while the batch is settling in the extractor. After cleaning, the filter cloths must be thoroughly scrubbed with a brush and warm water.)

Extraction:

Muriatic acid is added to the batch in lead lined extractor #6763 until it turns Congo Red paper blue or very black. Usually 15 to 20 pounds of 31% muriatic acid are required. The agitator in the extractor is turned off and 25 inches of benzol (770 pounds) measured in wall tank #6235 are added. The batch is agitated for 2 minutes and allowed to settle for twenty five minutes.

The benzol layer at the top is decanted by applying a vacuum no greater than 10 inches to the empty Monel still #9176 and opening the valves in the decanter line. The decanter arm is lowered as far as possible in the extractor so that no glycol appears in the sight glass. The valve under the sight glass is opened full and the flow in the decantation line is controlled by means of the valve above the sight glass. After the first benzol extract has been removed, 12 inches of benzol (365 pounds) are again dropped into the extractor. After stirring for 2 minutes, the batch is allowed to settle for twenty five minutes. The second extraction is removed as before and then the last 12 inches of benzol are added. After stirring for 2 minutes, the batch is allowed to settle for twenty five minutes and the last extraction is decanted.

Washing of Benzol Extract:

The vacuum on Monel still #9176 is released and any glycol on the bottom is drained and returned to lead extractor #6763. The level (i.e. the distance between the bottom of the hand hole and the top surface of the benzol extract) is approximately 10 inches. The agitator is turned on for one minute in order to mix the extracts. After the agitator is stopped, thirty gallons of fresh water measured by means of the water meter and one gallon of muriatic acid mixed with 2 gallons of water in a copper pail are added to the copper still and the batch is agitated for three minutes. After settling for twenty five minutes, the water wash on the bottom is drained to the sewer. Any muck is saved for separation in a bottle. The batch is washed a second and third time in the same manner using 30 gallons of water each time without any acid. All the clear water washes are also sent to the sewer.

Small amounts of glycol are removed by these washes and it is very important that the washing be efficient, otherwise the congealing point of the distilled trichlorophenol will be low. If the still were larger, the amount of wash water could be increased, and two washes would suffice.

Removal of Benzol:

The valves on the copper still are set for atmospheric distillation into receiver #8526. The agitator is started and the steam is turned on slowly until the benzol begins to distill (approximately 70°C still temperature). The steam

is then increased in order to distill the benzol as quickly as possible without boiling over the batch. When receiver #8526 is almost full, the distillate is switched to the underground tank. (The amount of benzol already in the underground tank should be noted on the log sheet, so that the total amount of benzol recovered from each batch can be determined. This is approximately 195 gallons.) The water is drained from receiver #8526 and is discarded. The benzol in this receiver is run into the underground tank. When the steam pressure has increased to 50 pounds (still temperature approximately 125°C) and the rate of distillation has slowed down, the distillate is switched back to receiver #8526 and vacuum is applied slowly until full vacuum (20 to 25 inches) is attained. Ten minutes after the benzol has stopped distilling at full vacuum and at a still temperature of at least 125°C, the vacuum and steam are turned off and the batch is cooled to 90°C with cooling water. The steam to the copper coil around the bottom discharge pipe is turned on in order to prevent the phenol congealing in the pipe line when it is drained. The crude is drained into a tared galvanized or tin lined drum and weighed. (A 4 oz. sample of the crude is obtained for the laboratory.) The average weight of crude is approximately 575 pounds.

The drum of crude is placed in building 68 hot box in order to keep it in a molten state.

Distillation of the Crude Trichlorophenol:

Three batches of crude are charged to the monel still #8867 by means of vacuum. (The operator must wear a face shield

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and gloves whenever molten trichlorophenol is being handled.) The steam to all the steam traced lines and to the hot water circulation tank is turned on. The hot water pump is started so that water at 70°C is circulated through the jackets of the Monel receivers, through the condenser and through the dephlegmator coil. The flow of water to the dephlegmator is adjusted to 5 gallons per minute as indicated by the rotameter. The three gas burners under the still are turned on full and cooling water to the vacuum traps is also turned on. The valves are set so that the first fraction distills into Monel receiver #8884 under coarse vacuum. Distillation usually starts when the still temperature is approximately 160°C and the vacuum is 20 mm. At this time the two inner burners are turned off and the outer burner is reduced to about a 5 inch flame. The steam supply to the hot water tank is reduced. After the first receiver is half filled, the distillate is switched to the other Monel receiver #8885 and the vacuum is changed from coarse to medium. All gas flames are turned off in the room and the benzol in the vacuum trap of the first Monel receiver #8884 is drained into a pail and is transferred to a drum outside the building. A pail is kept under the trap and the trap cooling water is turned off and the steam to the jacket of the trap is turned on to ten pounds. The bottom drain valve is rammed with a bent rod to insure all the benzol has been drained and then the gas flames in the room are ignited again. After the trap of the first receiver has been heated for ten minutes, the drain valve is closed, the steam turned off and the cooling water is started

through the jacket as before. The trichlorophenol in the first receiver which amounts to approximately 140 pounds is run directly into the light fraction drum. Distillation is continued at 5 to 8 mm. until the level of the molten trichlorophenol in the second Monel receiver #8885 can be seen in the level gage glass. The distillate is switched back to the first receiver and after one third of a pail of trichlorophenol is withdrawn from the second receiver, a 2 oz. sample is obtained. The congealing point and solubility of a few drops in 5% NaOH are determined on it. If the congealing point is 63.5°C or higher and it is clearly soluble in NaOH, the contents of the pail and receiver are run into the "B" drum. This drum should have a good galvanized or tin lining. If the congealing point is below 63.5°C , the sample generally is cloudy in the NaOH solution and the trichlorophenol is run into the light fraction drum again. The above procedure is repeated until the "B" cut is obtained. At this point the flow of hot water to the dephlegmator coil is reduced to one gallon per minute. During the "B" cut the receivers are filled to within one inch of the top of the level gage glasses, and the contents are run directly into the "B" drum. The oil bath temperature is between 250 and 270°C during this period.

After the second receiver has been drained, the cooling water to the corresponding trap is replaced with steam and the trap is allowed to drain into a pail. Usually it is necessary to preheat the drain valve with a steam hose before draining. After ten minutes, the steam flow is stopped and the cooling water is started again. Each time a receiver is drained,

the corresponding trap is heated. However, it is not necessary to drain the traps after the first two times as only a small amount of material collects in the bottom of the traps.

Near the end of the distillation, the distillation rate begins to slow down and the still temperature rises. From now on the receivers are filled only to the bottom of the level gage glass and the congealing point and solubility are determined each time the receiver is drained. As soon as the congealing point falls below 63.5°C , the trichlorophenol is drained into the "heavy fraction" drum. It may happen at this stage that the congealing point is low and the solubility is clear, but good trichlorophenol meets both criteria. Approximately 100 pounds of this fraction are usually obtained. The still temperature is not allowed to rise above 210°C , otherwise decomposition occurs in the still and the residue becomes hard and can be removed only by chopping and scraping. When the distillation is completed, the flames, hot water and steam supplies are turned off and the vacuum is released slowly. When the temperature in the still falls to 135°C , the residue is withdrawn by means of a suction drum. The residue which contains the monotrachlorophenyl ether of diethylene glycol is delivered into stock at no value. It must be remembered that after repeated heating the oil in the still jacket will polymerize and carbonize. This occurrence is usually indicated by an increasing temperature lag between both temperature and still temperature. Good bath oil generally shows a temperature lag of $60-65^{\circ}$; when the lag exceeds 110° , the condition of the bath oil should be checked and the oil renewed.

Process for 2,4,5 Trichlorophenol

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The vacuum traps are heated by means of steam and are completely drained.

The following data are obtained from a typical distillation:

Charge: 3 Batches of Crude - 1,754 lbs. total.

TIME	TEMPERATURE					VACUUM MM Hg	DEPHLEGMATOR			PRODUCT		RE- CEIVER #	REMARKS
	Still °C	Col- umn °C	Dis- til- late °C	Oil Jack- et °C	Circu- lating Water °C		Rota- meter gal/ min.	In- let water °C	out- let water °C	Frac. tion	C.Pt.		
5:30												Still char- ged. Burners on full.	
6:30	120	90	82	250	72	20	4.5	62	62			8884	Distilling
7:30	156	96	76	250	72	18	4.5	72	84	Light		8885	
8:30	158	130	82	265	74	12	4.5	66	95	Light	57.0	8885	
9:30	158	128	84	250	74	12	4.5	76	100	Light	61.0	8885	
10:30	150	130	88	275	74	12	1.0	80	100	B	62.0	8885	Switch Re- ceivers
11:30	150	130	84	275	74	12	1.0	80	100	B	62.6	8884	
12:30	150	132	84	275	74	12	1.0	78	100	B		8885	
1:30	150	128	82	275	74	5	1.0	78	100	B	64.2	8884	
2:30	156	132	88	275	74	5	1.0	70	100	B		8885	
3:30	158	132	84	280	74	6	1.0	70	100	B	62.3	8884	
4:30	184	130	98	285	84	9	1.0	80	100	Heavy	60.5	8885	
5:30	198	130	98	275	84	12	1.0	82	98	Heavy		8885	Heat off.

Light Fraction - 348 lbs. 19.8% of charge
 B - 1,118 lbs. 63.5% " "
 Heavy Fraction - 110 lbs. 6.3%
 Residue - 115 lbs. 6.5%
 1,691 lbs. 96.1%

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When sufficient quantities of light and heavy fractions have been obtained, they are distilled separately. Upon repeated distillation of the light fractions, a very light fraction is obtained which does not congeal at room temperature. This fraction which contains a large amount of 1,2,4 trichlorobenzene is delivered into stock at no inventory value.

Neutralization of Weak Ethylene Glycol:

Twenty pounds of solid soda ash are added to the empty steel neutralizer #6219. The glycol remaining in the lead lined extraction tank #6763 is transferred to the steel neutralizer by applying vacuum to the neutralizer. The sight glass in the transfer line is observed and as soon as the benzol layer appears, the flow is stopped by turning off the two valves under the extraction tank and releasing the vacuum in the neutralizer. The benzol in the pipe line between the bottom valve on the extraction tank and the valve next to the sight glass is drained into a pail and is returned to the extraction tank. The small amount of benzol in the extraction tank is allowed to remain there for processing with the next batch.

The agitator in the neutralizer is started and the glycol is neutralized by adding solid soda ash to a pH between 6 and 7 as determined by means of Hydrion paper. Approximately 10 pounds of soda ash are usually required. The level in the neutralizer at this time is 33" below the bottom of the hand hole (equivalent to 260 gallons).

The neutralized glycol is allowed to flow by gravity from the neutralizer into the four outside storage tanks #5640, 5641, 5642 & 9029.

Distillation of Weak Ethylene Glycol:

The glycol recovery still #8728 in building 54 is charged with a 28" level (distance between bottom of hand hole and top surface of charge) of weak ethylene glycol (400 gallons) from the outside storage tanks 5640, 5641, 5642 & 9029 by means of Westco transfer pump #8500 located in building 60. The starting switch for this pump is located near the still. Before pumping, the hand hole cover on the still is removed and the still is connected to the coarse vacuum pump #8656 so that no benzol fumes which might be present in the weak glycol will enter the room. After obtaining the exact level, the still is closed, the steam is turned on full and the valves are adjusted so as to distill into receiver #8670 under coarse vacuum from Nash pump #8656. The air bleed on the pump is opened wide so that a vacuum of 22 inches of mercury is obtained at the still. The cooling water is turned on full and the reflux regulator is set for total reflux. Distillation begins when the still temperature is approximately 75°C. The first fraction consists of weak isopropyl alcohol. Although the regulator is set for total reflux, product is obtained as only partial condensation occurs in the water tube condenser and the liquid cooler serves as both a condenser and cooler during the alcohol cut. The steam pressure maintained on the jacket of the still is limited by the cooling water supply. With the cooling water on full enough steam is used so that the distillate temperature is about 35°C. After 50 gallons of distillate have been obtained, all the alcohol has distilled and the water cut is obtained. The weak isopropyl alcohol (approximately 25 to 30% alcohol) is drained into a grounded drum outside the

building. During this period the reflux regulator is set for 1 to 1 reflux and the air bleed on the Nash pump is closed. The water cut is distilled first into #8671 receiver up to the 24" mark on the level gage glass (105 gallons) and then into #8670. When the distillation rate slows down, the vacuum is switched from the Nash pump to the tower coarse vacuum. The water in receiver #8671 is run to the sewer and the water obtained in the second receiver is saved in the recovered water tank #8599. At the end of the water cut the column temperature rises sharply. When it reaches 100°C and the vacuum is approximately 20 mm of mercury, the distillate which is now glycol is switched to empty receiver #8671 and the vacuum is changed from coarse to medium. The reflux regulator is set on zero and the valve in the reflux line is closed. At this time the cooling water is also reduced so as to maintain a 35°C distillate. Distillation is continued until the distillate slows down to a slow drip and the still temperature is above 150°C. The vacuum is released and any fluid residue is drained into a drum. (This residue contains about 35% glycol and is distilled in the same still when a sufficient quantity is available.) In general no fluid residue is obtained. The hot still is immediately washed with cold water from a 1½" hose using a full stream to dislodge the salts in the bottom of the still. This cleaning which usually takes forty five minutes must be very thorough or the following distillation will require more than ten hours.

Fifteen hundred to 1,700 pounds of glycol are obtained from each batch. Two batches can be run every 24 hours and in view of the fact that one still batch is equivalent to slightly more than 1½ batches of weak glycol, there is

Process for 2,4,5 Trichlorophenol

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sufficient capacity if no interruptions are caused by poor vacuum or cooling water supply.

The following data were obtained from a typical distillation:

Charge - 28" level (400 gals.)

TIME	TEMPERATURE			VACUUM		REFLUX	STEAM Pressure lbs/ sq. in.	PRODUCT	RECEIVER		REMARKS
	Still	Col- umn	Distil- late	Top of Column	At Receiver				#	Ht. Glass in.	
8:50											Still charged & started. Distilling
9:05	75				24		20		8671	0	
9:35	98	90	40		24	total	20	Weak IPA	8671	7	
9:45	104	82	40		26	1:1	40	Water	8671 (8670)	8	
11:15	106	80	42		26	1:1	70	Water	8670	24	
12:05	130	70	26		29	1:1	80	Water	8671		
12:45	134	100	26		30	0	90	Glycol	8671 (8670)	7½	Glycol Dis- tilling.
1:40	134	112	34	28	30	0	90	Glycol	8670	8	
2:50	132	112	30	18	30	0	90	Glycol	8670	22	
3:30	130	110	28	12	30	0	90	Glycol	8670 (8671)	30	Switch Rec.
4:20	130	112	28	10	30	0	90	Glycol	8671	0	
5:20	140	114	26	10	30	0	90	Glycol	8671	2	
6:20	150	122	24	10	30	0	90	Glycol	8671	3½	
6:50	152	122	26	10	30	0	90	Glycol	8671	4½	Heat off - Still Washed

Weak IPA - 387 lbs. - (27% IPA)

Ethylene Glycol - 1,525 lbs.

No Fluid Residue

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The following schedule lists the operations in building 54.

Hydrolysis

Equipment #8454

<u>Time</u>	<u>Operation</u>
7:30 A.M. - Batch C	Charge ethylene glycol, caustic soda flakes and tetrachlorbenzene.
8:15 A.M.	Heat on.
9:05 A.M.	Vent closed.
9:35 A.M.	At temperature.
1:35 P.M.	Begin to cool.
2:05 P.M.	Begin to blow batch to Bldg. 60.
2:30 P.M.	Batch blown.

Glycol Distillation

Equipment #8728

<u>Time</u>	<u>Operation</u>
8:20 A.M.	Charge 600 gallon still.
8:50 A.M.	Steam on.
9:05 A.M.	Distillation begins - alcohol cut.
9:45 A.M.	Water cut begins.
12:45 P.M.	Glycol begins to distill.
6:50 P.M.	Heat off - Drain.
6:55 P.M.	Washing still.
7:55 P.M.	Still clean - ready for charging.

Distillation of Crude
Trichlorophenol

Equipment #8868

<u>Time</u>	<u>Operation</u>
9:00 A.M.	Charging still.
9:30 A.M.	Heat on.
11:30 A.M.	Distillation begins.
9:30 P.M.	Heat off.
1:30 A.M.	Unloading residue

Process for 2,4,5 Trichlorophenol

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The following schedule lists the operations in building 60.

Neutralization

Equipment #6220

<u>Time</u>	<u>Operation</u>
8:00 A.M. - Batch B	Cooling batch to 25°C after neutralization.
8:45 A.M.	Start to filter.
9:15 A.M.	Neutralizer empty.
2:30 P.M. - Batch C	Next batch received from Bldg. 54.
3:00 P.M.	Start to neutralize after cooling.
4:00 P.M.	Neutralization completed - cooling to 25°C.

Filtration

Equipment #4653

<u>Time</u>	<u>Operation</u>
8:00 A.M. - Batch B	Add 50 gallons water to lead extractor.
8:45 A.M.	Start to filter.
9:15 A.M.	Filtration finished - blow press.
9:20 A.M.	First isopropyl alcohol wash on.
9:25 A.M.	Blow press.
9:50 A.M.	Clean press.

Extraction

Equipment #6763

<u>Time</u>	<u>Operation</u>
9:30 A.M. - Batch B	Making batch acid to Congo.
9:40 A.M.	25" Benzol added to extractor.
9:45 A.M.	Agitate and let settle for 25 minutes.
10:10 A.M.	Taking off extraction.
10:30 A.M.	First extraction off - second on.
10:35 A.M.	Settle for 25 minutes.
11:00 A.M.	Taking off second extraction.
11:05 A.M.	Second extraction off - third on.
11:10 A.M.	Settle for 25 minutes.
11:35 A.M.	Taking off last extraction.
11:40 A.M.	Last extraction off.
12:00 A.M.	Transfer weak glycol from lead extractor to steel neutralizer by vacuum.
12:15 P.M.	Extractor empty.

Process for 2,4,5 Trichlorophenol

Sec. V Page ²⁰~~19~~Neutralization of Weak
Ethylene Glycol

Equipment #6219

<u>Time</u>	<u>Operation</u>
11:55 A.M. - Batch B	Add soda ash to steel neutralizer.
12:00 A.M.	Start to receive weak glycol from lead extractor.
12:15 P.M.	Weak glycol in neutralizer.
12:30 P.M.	Neutralize glycol.
12:35 P.M.	Drain to outside storage tanks by gravity.
12:55 P.M.	Empty.

Benzol Extract Washing
and Distillation

Equipment #8528

<u>Time</u>	<u>Operation</u>
6:05 A.M. - Batch A	Benzol Starting to distill.
9:30 A.M.	All benzol off.
9:45 A.M.	Cooled to 90° and drained.
10:10 A.M. - Batch B	Begin to receive extractions.
11:45 A.M.	Drain glycol from bottom of copper still.
11:50 A.M.	Add first acid wash and agitate.
11:55 A.M.	Settle for 25 minutes.
12:20 P.M.	Draining first wash.
12:25 P.M.	Add second wash and agitate.
12:30 P.M.	Settle for 25 minutes.
12:55 P.M.	Draining second wash.
1:00 P.M.	Add third wash and agitate.
1:05 P.M.	Settle for 25 minutes.
1:30 P.M.	Drain last wash.
1:35 P.M.	Steam on still.
2:05 P.M.	Starting to distill.
5:30 P.M.	All benzol off.
5:45 P.M.	Cooled to 90°C - Draining.

Process for 2,4,5 Trichlorophenol

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The hydrolysis schedule in building 54 and the schedules in building 60 are repeated for each shift. During the week the schedule advances several hours so that the stills are finished and drained by 7:30 A.M. Saturday morning.

Process for 2,4,5 Trichlorophenol

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SECTION VI

HAZARDS OF PROCESS

Health

Caustic Soda Flakes:

Long rubber gloves and an eye shield must be worn by the operator when handling caustic soda flakes, as they produce painful burns upon coming in contact with the skin.

Muriatic Acid:

Long rubber gloves and an eye shield must be worn by the operator when handling muriatic acid.

Benzol:

When inhaled benzol acts as a nerve poison causing faintness and cyanosis. Benzol is also absorbed through the skin and produces the same symptoms as an inhalation. It can also produce dermatitis through the removal of the natural oils from the skin tissues. Benzol is also a cumulative poison and therefore, any small leaks from pipes or equipment must be taken care of immediately.

Trichlorophenol:

Molten trichlorophenol produces burns in contact with the skin, although the burns are slight compared with those produced by phenol and parachlorophenol. Nevertheless, long rubber gloves and a face shield must be worn whenever trichlorophenol is handled. Any spillages on the skin should be removed mechanically as much as possible and then the area should be bathe with 50% isopropyl alcohol and then thoroughly washed with soap and water.

Process for 2,4,5 Trichlorophenol

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Fire

Benzol has a low flash point of -11°C and precautions must be taken that no sources of ignition exist when benzol vapors are present. Whenever benzol is being transferred from or to a drum, the ground clamp and wire must be used.

The flash point of isopropyl alcohol is $+12^{\circ}\text{C}$ and therefore presents a fire hazard. The same precautions as indicated above for benzol should be taken.

Ethylene glycol has a flash point of 115°C and therefore presents a fire hazard at elevated temperatures.

Process for 2,4,5 Trichlorophenol

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SECTION VII

PRODUCTS AND BY-PRODUCTS

Trichlorophenol:

Congealing Point: 63.5°
 ~~62.0°~~ C minimum

Light Transmission: 90% minimum
(50% solution by
volume in 2B alcohol)

Delivered into stock in 55 gallon galvanized or tin
lined drums containing approximately 575 pounds.

Residue:

Delivered into stock at no inventory value in 75 gallon
black iron drums containing approximately 1,000 pounds.

This residue contains about 50% of the monotrchlorphenyl
ether of diethylene glycol.

Process for 2,4,5 Trichlorophenol

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SECTION VIII

COST DATA

Materials consumed per 100 pounds of Trichlorophenol

Distilled produced:

Tetrachlorbenzene	135 pounds
Caustic Soda Flakes	71 "
Ethylene Glycol	90 "
Muriatic Acid 31%	137 "
Isopropyl Alcohol 99% (with recovery)	25 "
Benzol	20 "
Soda Ash	6.8 "

Labor required

To operate three shifts, ⁶7 men are required. One hydrolysis batch is made per shift and with a yield of 74%, 444 pounds are obtained from each batch,

therefore $\frac{7 \times 8}{3 \times 444/100} = 4.2$ man hours per 100 pounds of
Trichlorophenol Distilled
(no recovery of IPA)

Allowing 10% more for night supervision, repairs, cleaning, and rectification of recovered isopropyl alcohol, a labor requirement of 4.6 man hours per 100 pounds of trichlorophenol is obtained.

Process for 2,4,5 Trichlorophenol

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SECTION IX

LITERATURE AND RESEARCH SOURCES

Conversion of 1,2,4,5 Tetrachlorbenzene into 2,4,5 Trichlorophenol by W. S. Gump, Dec. 29, 1945.

2,4,5 Trichlorophenol Process by H. G. Krebs, April 17, 1943.

2,4,5 Trichlorophenol (Ethylene Glycol Process) by H. G. Krebs, Dec. 12, 1947

Trichlorophenol File in R.L. 3.

The Toxicity and Potential Dangers of Ethylene Glycol by Wiley, Hueper and vonOettingen, Journal of Industrial Hygiene and Toxicology, Feb. 1936.

Dow Glycols - Published in 1947 by the Dow Chemical Co.

Crosby-Fiske-Forster Handbook of Fire Protection.

Poisons, Their Properties, Chemical Identification, Symptoms and Emergency Treatments by J. Brookes and H. N. Alyea.

Process for 2,4,5 Trichlorophenol

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SECTION X

DRAWINGS

Tracings of the following drawings pertaining to the process are on file in the Engineering Department:

<u>Description</u>	<u>Equipment No.</u>	<u>Drawing No.</u>
500 Gallon Steel Neutralizer	6219	B-3072
750 Gallon Steel Recovered Water Storage Tank	8599	C-2861
750 Gallon Steel Recovered Benzol Storage Tank	8600	C-2861
150 Gallon Steel Receivers	8670 & 8671	C-2890
18" Monel Column for Monel Still	----	B-2912
185 Gallon Monel High Vacuum Still	8868	2899
25 Gallon Vacuum Receiver	8526	C-2846
Gooseneck for Monel High Vacuum Still	----	C-3008
600 Gallon Steel Recovery Still	8728	B-2862
Stainless Steel Pan for Filter Press #4653	5306	C-2948
Location of Outlets on Water Tube Steel Condenser	8767	C-2971
Reflux Preheater for 600 Gallon Still	8753	D-2895
1,130 Gallon Lead Lined Extraction Tank	6763	2256
1,130 Gallon Lead Lined Extraction Tank Coil	----	B-2850
Circulating Water Tank for Monel Still	----	C-2918
Liquid Cooler for Glycol Still	----	B-2897
Instrument Panel for 600 Gallon Still	----	2900
30" Column for Glycol Still	8787	B-2865
Detail of Flanges for 30" Column	----	C-2866
Monel Coil Condenser for Monel Still	8824	2061
25 Gallon Monel Receivers	8884 & 8885	C-2909
250 Gallon Stainless Steel Neutralizer	6220	2856
Gas Burner Support for Monel Still	----	C-2934
250 Gallon Hydrolysis Kettle	8454	B-2733-1

Process for 2,4,5 Trichlorophenol

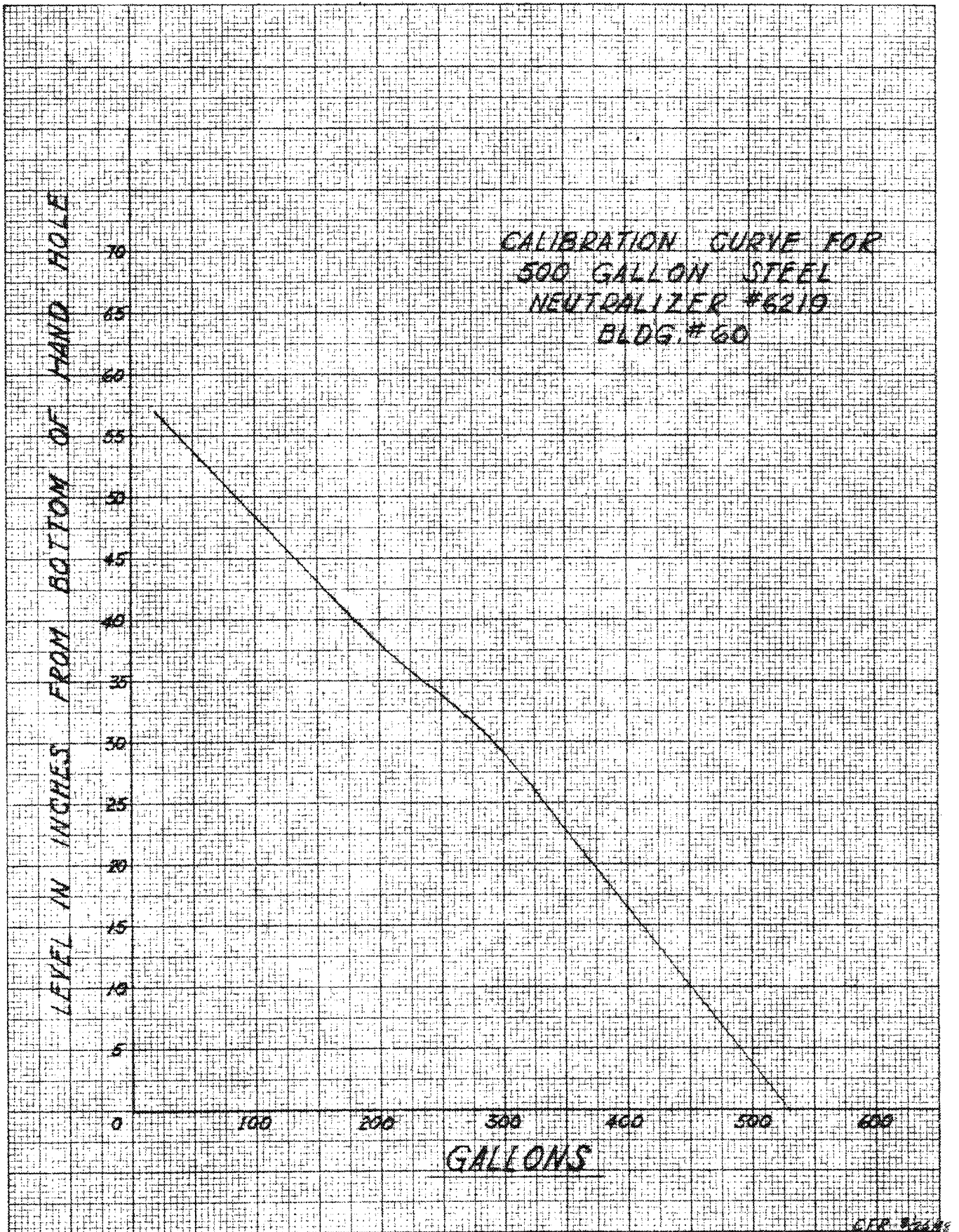
Sec. X Page 2.

250 Gallon Hydrolysis Kettle Details	----	A-2738-2
350 Gallon Copper Still	8528	B-2852
Assembly of Sigmamotor Pump and Motor	8858	A-3017
75 Gallon Alcohol Tank	8565	2028-A
Installation of Neutralizer #6219, Pump #8500 and 4 - 275 Gallon Steel Storage Tanks	----	A-3078
250 Gallon Benzol Feed Tank	6235	2255
Decanter on Lead Lined Extractor	----	2414
T.C.P. Process Flow Sheet	----	C-2812
2,4,5 T.C.P. Process Bldg. #54 West Wall	----	2839
2,4,5 T.C.P. Process Bldg. #60 West Wall	----	2843

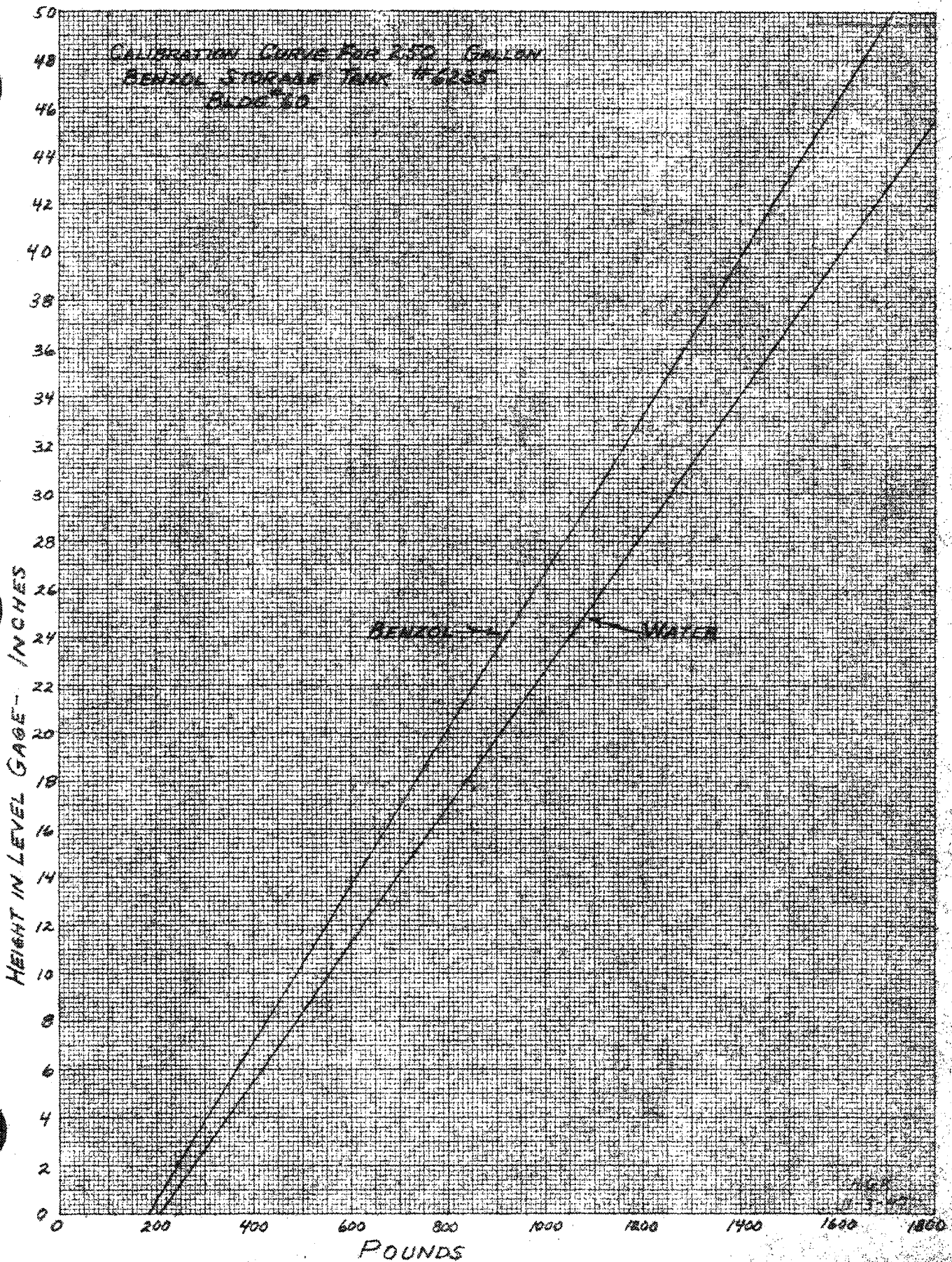
LIST OF GRAPHS AND DRAWING
CONTAINED IN THIS PROCESS PROCEDURE

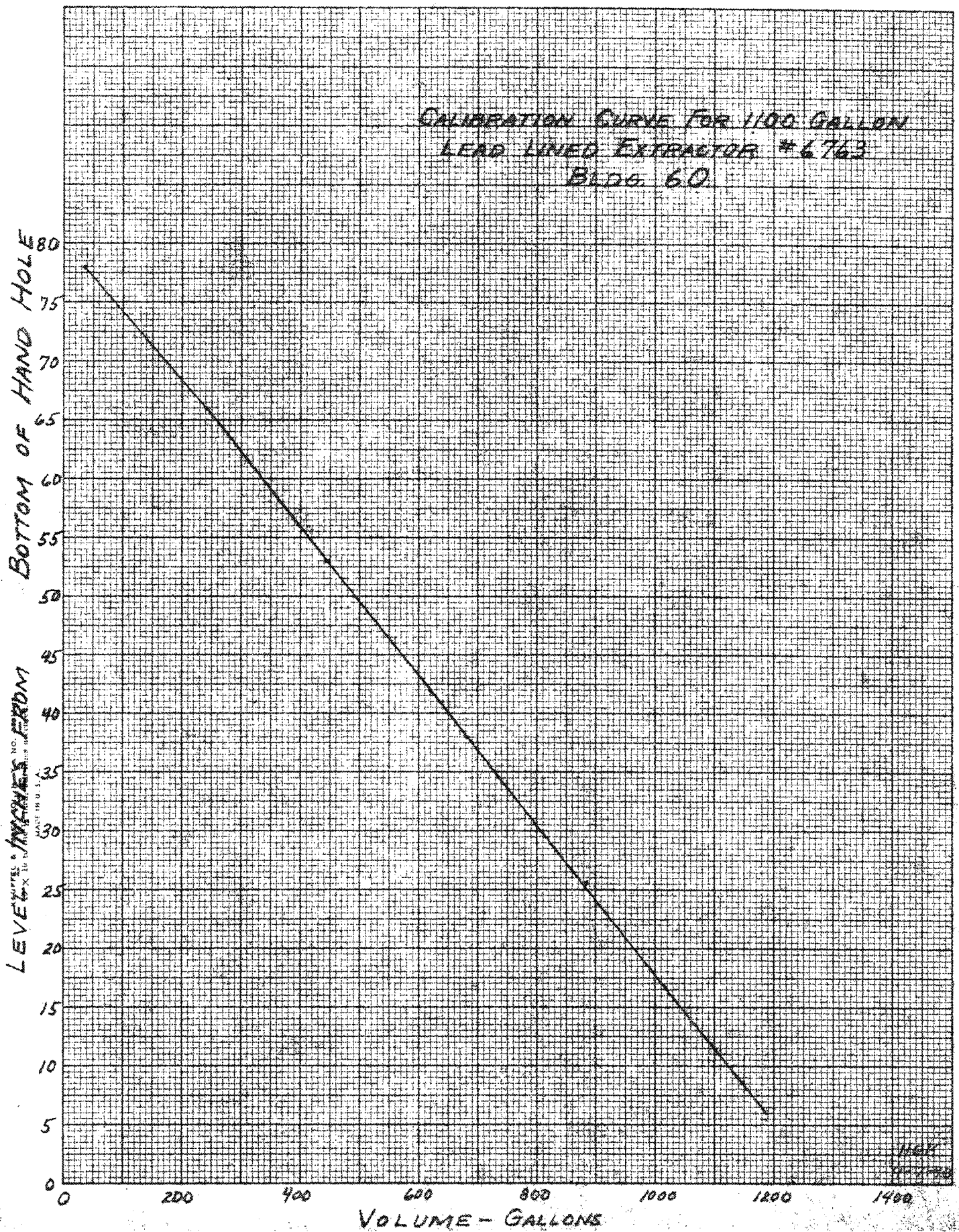
Calibration Curve for 500 gallon Steel Neutralizer	#6219
Calibration Curve for 250 gallon Benzol Storage Tank	#6235
Calibration Curve for 1,100 gallon Lead Lined Extractor	#6763
Calibration Curve for 25 gallon Vacuum Receiver	#8526
Calibration Curve for 350 gallon Copper Still	#8528
Calibration Curve for 600 gallon Steel Still	#8728
T.C.P. Process Flow Sheet	C-2812

10 X 10 TO USE IN 1970. OUR LISTS APPROVED.
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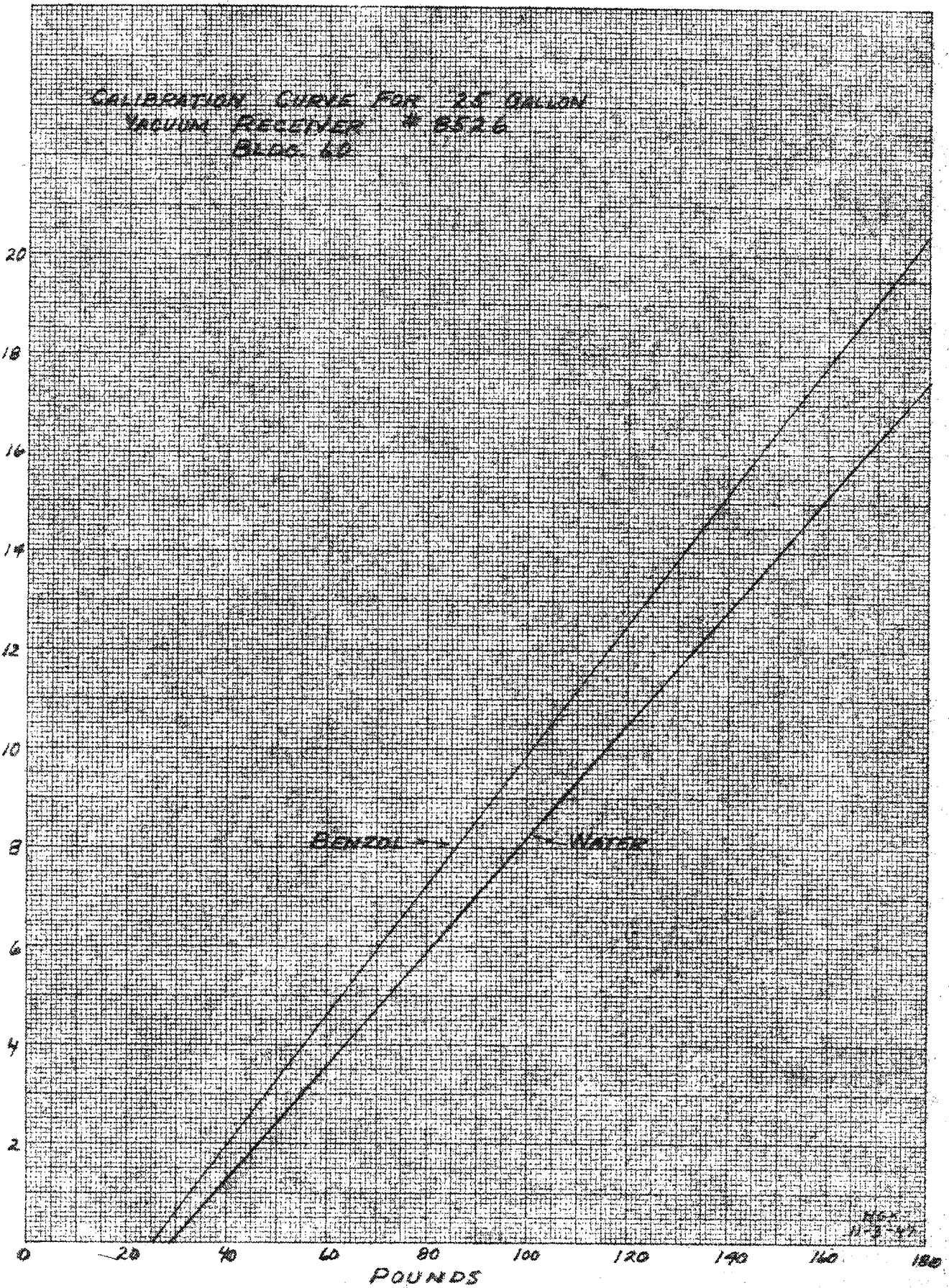
KEUPPEL & KESER CO., N. Y. NO. 3094-14
 MILLINER, 501, 10th Avenue, N. Y. City
 MADE IN U. S. A.

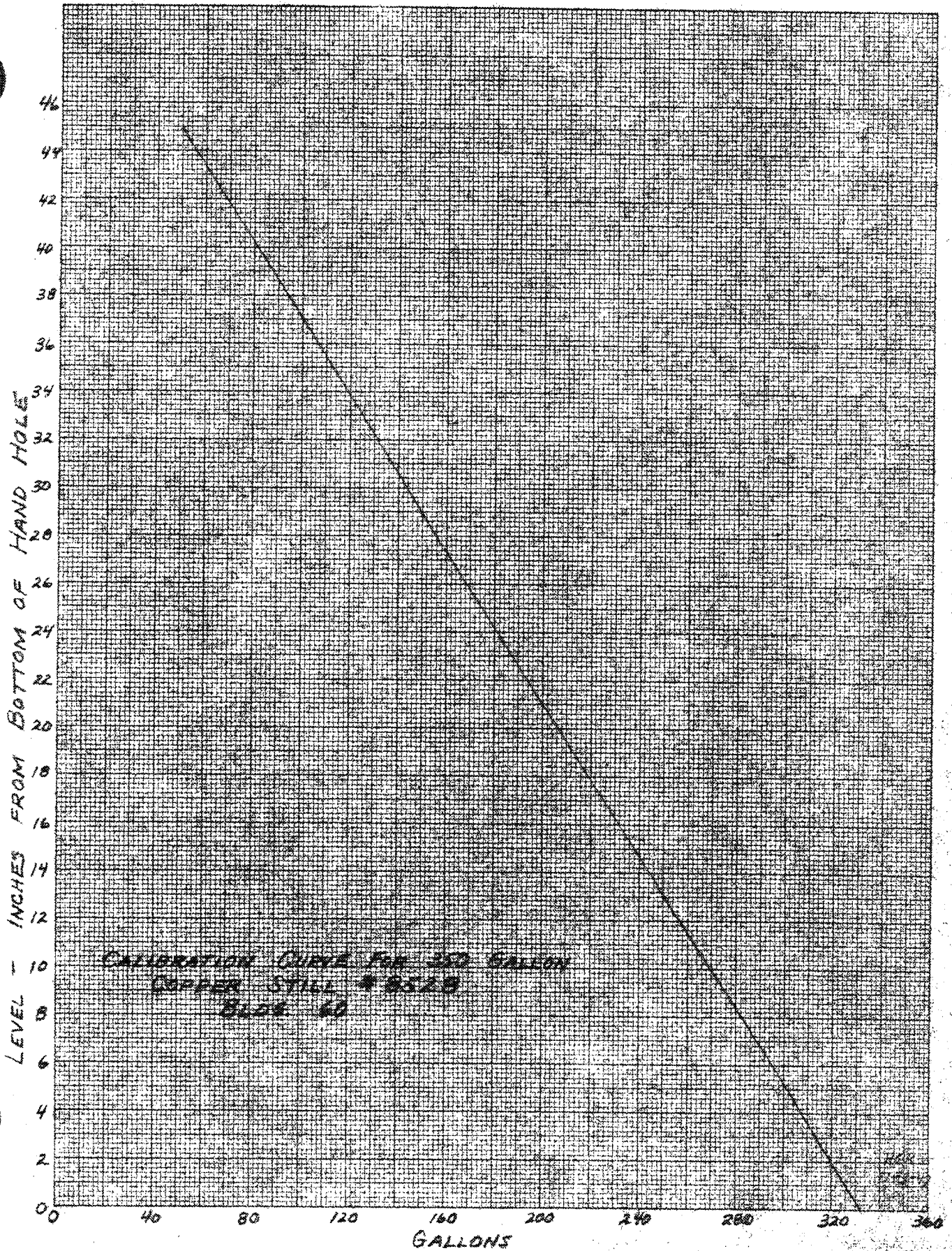




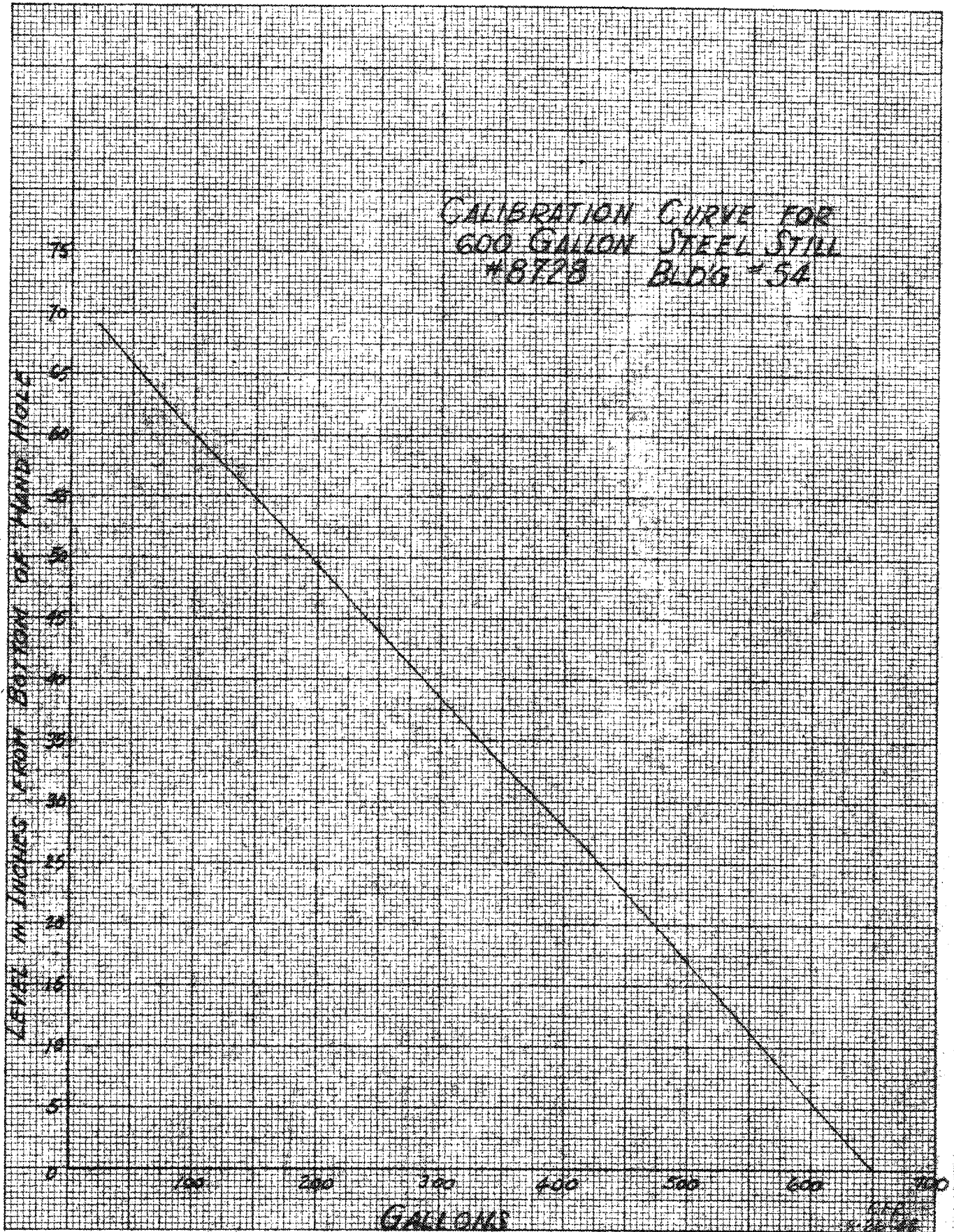
KEUFFEL & ESSER CO., N. Y. NO. 3791-14
 MILLIGAN, A. 1915, from accepted, cm. line heavy
 MADE IN U. S. A.

HEIGHT IN LEVEL GAGE - INCHES





KEUFFEL & ESSER CO., N. Y. NO. 586114
MILWAUKEE, 3 mm. fixed standard, on fibrous base
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KALUFINE & SONS CO., N. Y. NO. 8951-12
1 1/2" X 10" to the 1/2" inch, 50th lines standard.
MADE IN U. S. A.